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THREE STAGE BLEACHING OF RECYCLED FIBERS CONTAINING 30% GROUNDWOOD

by

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ABSTRACT

A three stage bleaching sequence for a mixture of recycled chemical and groundwood fibers was investigated for maximum brightness increase. The three stages consisted of a reducing or color stripping stage, an oxidizing stage, and last a mild alkaline extraction. The variables in this thesis are pH conditions in each stage and bleaching agent concentrations for each stage. At more alkaline pH's and higher concentrations of hydrogen peroxide there was the greatest increase in brightness. The was no observed effect of altering the concentration of the reducing agent.

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INTRODUCTION

Paper with high brightness has always been one of the demands of the consumers. Recently therehas been a priority placed on wood conservation and more recycling of papers.[11] The injection of recycled fibers (secondary fibers) into the virgin supply has confronted the pulp and paper industry with many new problems in strength and optical properties of the finished sheet.[12]

This thesis concentrates on the optical properties with respect ot bleaching of recycled fibers. The objective is to find the optimum conditions in a three stage bleaching process to increase the brightness as well as enhance other optical properties of a mixture of recycled chemical and groundwood fibers.

Three main varibles, the chemical concentration in the first stage, the chemical concentration in the third stage and the pH at which the reactions occur constitite the basis for analysing data in order to find the optimum conditions for brightening. The main objective of the bleaching is, first to remove any residual lignin in the chemical pulp, and a small percentage from the groundwood pulp. Removal of a medium to large percentage of lignin from the groundwood will reduce the yield which is undesirable. Second is to remove any coloring agents such as dyes, toners, and selective ink pigments.

HISTORICAL REVIEW

Bleaching of recycled fibers have been in practice since the beginning of the 1900's. However there has been no significant studies or advancements of this area since that time. Thus historical literature pertaining to bleaching of virgin fibers will be reviewed.[11]

In 1774 chlorine gas was discovered and found to be a new and very effective bleaching agent with respect to wood pulp.[19] During the passage of time derivatives formed from chlorine gas were found to act as bleaching agents. These included potassium, sodium, and calcium hypochlorite. It was not until the 1920's and 30's that chlorine dioxide was discovered to act as a bleaching agent.[19] It was brought in commercially in the 40's. World War 2 saw the evolution of peroxide which eventually was used in the paper industry for bleaching.[9,19] Reducing agents for bleaching, such as sodium borohydride were commericalized in the 1950's.[20]

Up until the end of the 19th century all bleaching was done in one stage. In the 1920's multistage bleaching was first carried out.[19] Since the 20's multistage bleaching processes have expanded with the development of new and more efficient methods.

In the literature search performed there has been no direct historical account of bleaching with respect to recycled fibers. It is indeed evident that bleaching of recycled fibers has been investigated by the numerous patents and few paragraphs directed to recycled fibers, which have been reviewed.[23,13] These patents give the variables and components necessary to bleach certain types of recycled fibers. Mention of the bleaching of recycled fibers in texts or articles refers to the need of futher research in this area. Thus, this report endeavors to contribute and provide further incentive to continue the study of bleaching recycled fibers.

THEORETICAL DISCUSSION

The theory behind bleaching and how it affects the pulp system is given in great detail by Holst[15] and reviewed by Browning[3]. The color of substances (mainly organic) is associated with mobile pi electrons in a system of conjugated double bonds. When the number of carbon conjugated double bonds is small in the main carbon chain, light is absorbed in the UV spectrum range. As the number of carbon conjugated double bonds increases, the length of the main carbon chain increased, thus causing light to be absorbed into the visible spectrum range which causes the yellow color to appear. Groups which cause conjugated double bonds are carbonyl, phenyl, quinone, and adjacent carbonyl groups. Bleaching therefore breaks these conjugated double bonds and demobilizes the pi electrons which are engaged in the absorbtion of light in the visible spectrum due to excitation. It is known that many colored substances that are of quinoid nature have conjugated double bond systems which sometimes appear in recycled material.

Pulping and bleaching of virgin fibers are considerably different than that of recycled fibers. The cooking chemicals, pH, temperature, and time relationships vary from virgin to recycled fibers, but the bleaching agents used do not differ significantly. Thus a relationship of bleaching chemicals and variables must be derived from research based on bleaching of virgin fibers. This information is applied to bleaching of recycled fibers due to little information written in this area.

In recycled fibers there are dyes, coating agents, and ink pigments that are bleach comsuming agents.[21] These must be handled in a way different from virgin fibers which do not contain these agents. A common problem in the "yellowing" of the pulp in virgin and recycled fibers, its colored functional groups. It is observed[14,21] that carbonyl, carboxyl, and aldehyde groups are the most responsible for yielding the "yellowing" color of pulp and causing color or brightness reversion. Other less significant causes are resins, impurities, and other functional groups attached to the cellulose.[14] Still another problem of bleaching is cellulose degradation due to high pH or very low pH. Consideration of pH, temperature, and chemicals used should be controlled to prevent degradation.

CHLORINE DIOXIDE

Over the past years, experience, and research have shown chlorine dioxide to be more efficient and economical than hypochlorite or chlorine gas in a five stage bleaching of kraft pulp.[10] This also applies to some virgin bleaching processes. The use of chlorine dioxide instead of hypochlorite before the peroxide stage reduced the peroxide requirment.[4] Chlorine dioxide degrades cellulose to a lesser extent then hypochlorite.

A statistically designed experiment showed replacing chlorine with chlorine dioxide in a five stage bleaching sequence did save in the use of alkali chemicals in the extraction stage and reduced bleaching costs.[10] Chlorine dioxide is most effective as a bleaching agent when the pH range is 3-5, the temperature is 60-80°C, and pulp consistancy ranges from 6-18%.[19,22] It is mainly used as a lignin removing agent.

Since 70% of the pulp under study will be chemical pulp it is assumed some lignin still remains even after bleaching in the virgin stage. This lignin should be removed effectively by the use of a low concentration of chlorine dioxide. Using this low concentration should not adversly affect the yield of groundwood pulp by removing a large percentage of lignin.[19] Chlorine dioxide will be used in the first stage bleaching because its potential to remove any lignin present, mild degradation on cellulose fibers which will display strength loss due to reprocessing, and its advantages over chlorine and hypochlorite mention above.

HYDROGEN PEROXIDE

Since the introduction of hydrogen peroxide into the paper industry in the late 40's and early 50's[8] it has been applied in many segments of the bleaching process. Some advantages of hydrogen peroxide are improved brightness, good stability of color to heat in the drying process, better absorbency to water and oil, and very little pulp shrinkage or yield reduction.[21.22] Hydrogen peroxide tends to bleach out selective azo dyes present in the pulp.[7] Literature cited hydrogen peroxide to increase brightness 8-10 points after the chlorine dioxide stage and of groundwood pulp alone.[9,22,3,16,4,6] Optimum conditions for bleaching groundwood are pH range of 10.5-11, temperatures ranging from 60° to 90°C, consistancy of 4-10%, time 1.5-2.0 hrs., and use of 1% hydrogen peroxide based on B.D. fiber.[3,22] Hydrogen peroxide affected kraft pulp viscosity approximatly 1cp. This is not dependent on the concentration of the peroxide but is seen to be a consistant decrease.[4] Hydrogen peroxide will be used due to its increase of brightness in groundwood and after chlorine dioxide bleaching. It also has good potential for removing some azo dyes which are present in recycled pulp. Also little yield reduction and improved brightness stability are good properties needed when bleaching recycled fibers.

HYDROGEN PEROXIDE MIXED WITH CHLORINE DIOXIDE

Nothing in literature has revealed that mixing chlorine dioxide with hydrogen peroxide, acting as one bleaching agent, has ever been done. This combination will be tried experimentally on the following argument. Optimum bleaching with hydrogen peroxide occurs at a pH of 10.5-11. As bleaching progresses, the pH decrease from 10.5 to about 6.5-7.0.[24] Under controlled conditions the final pH can be brought to 5 which happens to be the optimum pH for bleaching with chlorine dioxide. If no side reactions occur preventing the formation of the active bleaching agents then this step could prove to be economical in cost, chemical consumption, and time.

In alkaline medium Rydholm observes that the oxidation power of chlorine dioxide ion is depressed.[19] As the pH decreases the oxidation power increases. Thus the chlorine dioxide would not take effect as an active bleaching agent until the peroxide has reacted enough to bleach the pulp in part and reduce the pH.

EXTRACTION

The extraction stage with a caustic solution is used to remove oxidized cellulose, resinous substances, and chlorinated lignins. The removal of these components proves benefical after use of chlorine, chlorine dioxide, or hypochlorite in increasing the brightness.[21] These components act as bleach consuming agents as well as hindering bleaching action in the next stage. Thus when removed, brightness increases several points and reactions times are reduced.

REDUCING AGENTS

One of the main characteristics of reducing agents mainly zinc hydrosulfite as an bleaching agent is the removal of various dyes. Barton[1] gives an extensive account on the classification of dyes which can be permanently and partially decolorized by zinc hydrosulfite. It has also been found that zinc hydrosulfite does not affect pulp strength to any extent, is more economical than other reducing agents, and removes a wider range of dyes.[21] Conditions for use of zinc hydrosulfite are a pH range of 4-7, a temperature range of 140-180°F, percent consistancy of 3-4, concentration of chemical 1-4% based on B.D. fiber, and standing time of 1.0 hr.. It is stressed that atomspheric oxygen not be allowed to enter the bleaching system during agitation.[2,26] The oxygen reacts with the reducing agent to break it down or oxidize it into a non-effective bleaching agent. Rapson[18] shows that peroxide and reducing agents used individually increased brightness of nine groundwood species. This was also evident when a reducing agent followed a peroxide stage.

There have been indications of sodium borohydride being used more and more for a reducing agent in multistage bleaching.[17,20,21] Sodium borohydride causes reduction of carbonyl groups to alcohol groups, and improved reflectance over a wider range than sodium or zinc hydrosulfite.[17]

Zinc hydrosulfite will be used in the experiment due to its low cost, effectiveness in removing a wide range of dyes inherent in recycled paper, and the small effect it has on the pulp strength.

SEQUESTERING AGENTS

Both peroxide and reducing agents degrade rapidly when they come in contact with metal ions. Sodium silicate in the peroxide and EDTA, or sodium tripolyphosphate in the reducing agent will complex with the metal ions. This action preserves the bleaching agent and increase brightness 1-4 points.[1,26]

WASHING

Washing is vital after the bleaching process is done. Washing removes residual bleaching chemicals, sequestering agents, and any by-products formed during bleaching, It is the removal of these constitutants that prevents color deterioration, and reoxidation of any solubilized dyes or chemicals.[21,25] It was shown that a washing step after each stage in a five stage bleaching of kraft pulp yielded no significant difference in brightness when compared to pulp that was not washed and contained residual bleaching chemicals. [27] Therefore a washing step after the final bleaching stage would be sufficient to remove any residual chemicals in order to prevent color reversions and side reactions. This yields to water conservation and reduced time for the bleaching process.

PROJECT OBJECTIVE

A three stage bleaching process was developed for recycled fibers. The first stage bleaching consisted of sodium hydrosulfite for removal of dyes, coloring agents, and increased brightness stability. This stage acted as a color stripping stage. This has been indicated as being benefical in lowering bleach demand by removal of various coloring agents.[26]

The second stage consisted of a mixture, at various concentrations of hydrogen peroxide and chlorine dioxide. This stage was incorporated for removal of lignin, dyes, and other coloring agents not affected by the first stage. The third stage of mild alkaline ectraction is last in order to remove solubilized lignin, dyes, and residual chemicals. Wash steps with hot water was used between each bleaching stage to remove residual constituants in the pulp sample.

Graphs and tables are used in order to interpretate the data results.

EXPERIMENTAL PROCEDURE

Materials: newsprint, used IBM computer paper, and chemical office bond paper that was run through a A.B. Dick offset press were used to make up the recycled stock.

Chemicals: the following chemicals used are given with respect to their use in each bleaching stage: Stage one, the reducing or color stripping stage, sodium hydrosulfite ($Na_2S_2O_4$), sodium tripolyphosphate ($Na_5P_3O_{10}$).

Stage two, the oxidizing stage, hydrogen peroxide, a 3% solution made from stabilized 30% H_2O_2, chlorine dioxide solution (ClO_2) made from dissolving sodium chlorite in an acidic solution, sodium silicate (Na_2SiO_2).

Stage three; mild alkaline extraction, sodium bicarbonate ($NaHCO_3$). Chemicals used for pH adjustments were sodium hydroxide (NaOH), and hydrochloric acid (HCl).

Equipment: the equipment necessary to carry out the bleaching method are; four polyethylene bags, six liter capacity, a hot water bath, two four liter stainless steel beakers, one four liter waring blender with three speeds, suction filtration flask with 15cm buchner funnel, pH meter, bunsen burners, thermometers °C, and stainless steel spatulas.

Procedure: an sufficient quantity of pulp was prepared by using the recycling unit at Western Michigan University. Sufficient pulp of chemical and groundwood content for the entire studies was run through the hydropulper, belcor, finchk screen, bird cyclone cleaners, bergstrom reverse cleaners, side hill washer, and the screw press. Appendix A contains all experimental data pertaining to the use of the recycling unit.

A statistical block and treatment diagram is used to lay out the experimental procedure. The treatments contain the pH variable and the blocks contain the bleaching agent concentration variable. Bleaching agent concentrations are based on O.D. pulp used, in this case 60g O.D.. Below are the block and treatments used.

Treatments	1	2	3
pH stage 1:	7	5	4
pH stage 2:	10	7	5
pH stage 3:	8.5	8.5	8.5

Blocks	A	B	C
Ratio H_2O_2/ClO_2	.316/.110	1.05/.0551	2.11/.0165

Values are given as % active Cl_2.

There are two charts or block and treatment diagrams where each chart represents a different concentration of the reducing agent in stage one.

Chart	1	2
% $Na_2S_2O_4$	2.21	.551

All percentages are based on O.D. pulp. Two trials for each cell in the block and treatment diagrams were run for reproducability. Consistancys of the pulp samples ranged from 3.44 to 4.25%.

The systematic steps for each individual run is described as follows; the first stage is the reducing stage where the pulp and hotwater bath are brought to 60°C. The pulp is then added to the blender along with the desired amount of sodium hydrosulfite and sodium tripolyphosphate and adjusted to the proper pH. Blend for one minute on low speed and put into polyethylene bag and let set 1.5 hours in the hot water bath. Pulp sample is washed with hot tap water using suction filtration.

The pulp sample is then heated to 80°C and put into the blender. The desired concentrations of hydrogen peroxide chlorine dioxide and sodium silicate are added and adjusted to proper pH. Blend pulp slurry on low speed for one minute and then put in a polyethylene bag and sit in hot water bath at 80°C for 1.5 hours. Pulp samples is again washed with hot tap water by suction filtration.

Finally the pulp slurry is heated to 45°C and put into the blender. Sodium bicarbonate is added to bring pH to approx. 8.5 and mixed at low speed for one minute. The slurry is then added to the polyethylene bag and sit in the hot water bath at 45°C for one hour. The sample is washed as above.

Six 2.5g handsheets were made from each trial using the

Noble and wood sheet mold machine.

EXPERIMENTAL RESULTS

The data of the pulp pertaining to the recycling unit is shown in data set 1. In this data set ash values were obtained from pulp samples located at the hydropulper and at the screwpress. Fiber classification was done according TAPPI standards for the Bauer Mcnett classifer on samples at the hydropulper and screwpress. Brightness and opacity values are given for samples at the hydropulper and screwpress.

Data sets 2 and 3 give the brightness and opacity values respectivily for each cell in the block and treatment diagrams. Values were obtained from averaging the two duplicate trial runs together. The differences in birghtness and opacity values between the duplicate runs are given in appendix B.

The increase in brightness of each trial compared to the intial value are also presented in data set 2 and graphs 1-4. The decrease of opacity (except one case) compared to the intial opacity is given data set 3.

DISCUSSION OF RESULTS

A) Processing through the recycling unit:

It was observed that the percentage ash of the recycled pulp dropped 1.86 percent after processing through the recycling unit.

This clearly indicates that the total combined operations of the unit was effective in seperating ink pigment, fillers, coatings and other inorganics from the fiber stock. The 1.56 point increase in brightness comparing intial pulp to final pulp gave some evidence that possibly ink pigments were removed from the groundwood and chemical papers.

There was a large quantity of fiber fines (34% by weight) as determined by the Bauer Mcnett fiber classification of the pulp slurry at the hydropulper. Taking a pulp sample at the screwpress after recycling the pulp we find the sample contains only 11.4% by weight of fiber fines. This indicates that the pilot plant recycling process caused a loss of 23.0% by weight of fiber fines.

B) Three Stage Bleaching:

Reviewing data set 1 and graphs G1-G2 we see that as the pH goes from an acidic condition in stages one and two to an alkaline condition the brightness increases. This also was the case when a different concentration of sodium hydrosulfite was used. The optimum pH in this bleaching process would then be treatment one, where stage one was pH 10, stage two pH 7 and the third stage pH 8.5.

Looking again at data set 2 and G3-G4 the brightness increased from 68.9 to 74.4 as the ratio of H_2O_2/ClO_2 becomes larger. This clearly shows that hydrogen peroxide at higher concentrations than ClO_2 and at alkaline pH conditions was the optimum set of conditions for the bleaching process in question.

There is no substantial evidence to conclude that the mixture of hydrogen peroxide and chlorine dioxide was effective as an micture. Under alkaline conditions of pH 10 the bleaching in stage two decreased only to a pH of 9.0 from 10.0 thus not lowering the pH to an acidic level in order to allow chlorine dioxide to take on its full oxidizing power. The reverse was also true, at acidic conditions in stage two (pH5) the bleaching in this stage did not move to the alkaline condition, only to pH 4.5 thus slightly more acidic. This did not allow hydrogen peroxide to become an effective oxidizing agent.

Reviewing data set 3 we see in the treatment columns 1-3 that opacity in general was decreased a pH conditions moved to the alkaline side. There is not enough data to show that one concentration of the reducing agent sodium hydrosulfite is more effective in increasing the brightness than another. In data set 2, chart 1 had fOur bleaching trials higher in brightness increase than chart 2. Chart 2 had five bleaching trials that were higher in brightness increase than chart 1. In each case the brightness increase or decrease between the charts was very small. The above also is true for opacity decrease with respect to different concentrations of sodium hydrosulfite.

CONCLUSIONS

Recycling the mixture of chemical and groundwood pulp through W.M.U. recycling unit was effective in increasing the birghtness, and reducing the percent ash, but a loss of fiber fines to the system is unavoidable.

For maximum increase in brightness a reducing, oxidizing, and mild alkaline extraction sequence was followed. The reducing stage at a pH of 7 using a 2.21% solution of sodium hydrosulfite at a bleaching time of 1.5 hours was followed. The oxidizing stage used a pH of 10 and a ratio of hydrogen peroxide to chlorine dioxide concentration of 2.11%/.0165% at a reaction time of 1.5 hours. The last stage of mild alkaline extraction uses sodium bicarbonate to buffer the pulp slurry ot pH 8.5 with a one hour retention time.

RECOMMENDATIONS

Further study into bleaching of recycled fibers may include analysis on ink pigments, dyes, and toners and how they may best be removed from a pulp slurry. Investigation of optical properties between each bleaching stage would be benefical in indicating which stage is the most effective in the brighting of pulp.

DATA SET 1

	Hydropulper	Screwpress
% Ash	2.00	.147
Brightness	65.9	67.5
Opacity	88.0	88.7

Fiber Classification

HYDROPULPER

Screen Mesh					
Wt. fibers %	.675	27.8	22.3	14.9	34.4

SCREWPRESS

Wt. fibers %	.881	31.4	34.0	22.3	11.4

DATA SET 2

Chart 1 (2.21% $Na_2S_2O_4$)

	1	2	3
A	71.4	70.1	68.9
B	73.1	71.3	69.8
C	74.4	71.7	70.8

Brightness Values*

	1	2	3
A	3.96	2.56	1.39
B	5.63	3.85	2.28
C	6.90	4.22	3.35

Brightness Increase

Chart 2 (.551% $Na_2S_2O_4$)

1	2	3
71.3	69.6	68.9
73.3	71.5	70.2
73.9	72.0	68.4

Brightness Values*

	1	2	3
A	3.86	2.10	1.46
B	5.82	4.00	2.56
C	6.39	4.52	0.94

Brightness Increase

ments	1	2	3
age 1	7	5	4
age 2	10	7	5
age 3	8.5	8.5	8.5

Blocks	A	B	C
H_2O_2/ClO_2	.316/.110	1.05/.0551	2.11/.0165

Values given as % act. Cl_2

* Values are the average of the two trial runs.

DATA SET 3

Chart 1 (2.21% $Na_2S_2O_4$)

	1	2	3
A	87.4	88.3	87.2
B	86.2	87.9	88.1
C	86.5	87.8	87.5

Opacity Values*

	1	2	3
A	1.26	.360	1.46
B	2.46	.760	.560
C	2.16	.860	1.16

Opacity Decrease

Chart 2 (.551% $Na_2S_2O_4$)

	1	2	3
A	86.7	87.5	89.6
B	86.1	88.7	88.2
C	86.9	87.8	88.7

Opacity Values*

	1	2	3
A	1.86	1.16	-9.40
B	2.56	0.00	.460
C	1.76	.86	0.00

Opacity decrease

Treatments	1	2	3
stage 1	7	5	4
stage 2	10	7	5
stage 3	8.5	8.5	8.5

Blocks	A	B	C
H_2O_2/ClO_2	.316/.110	1.05/.0551	2.11/.0165

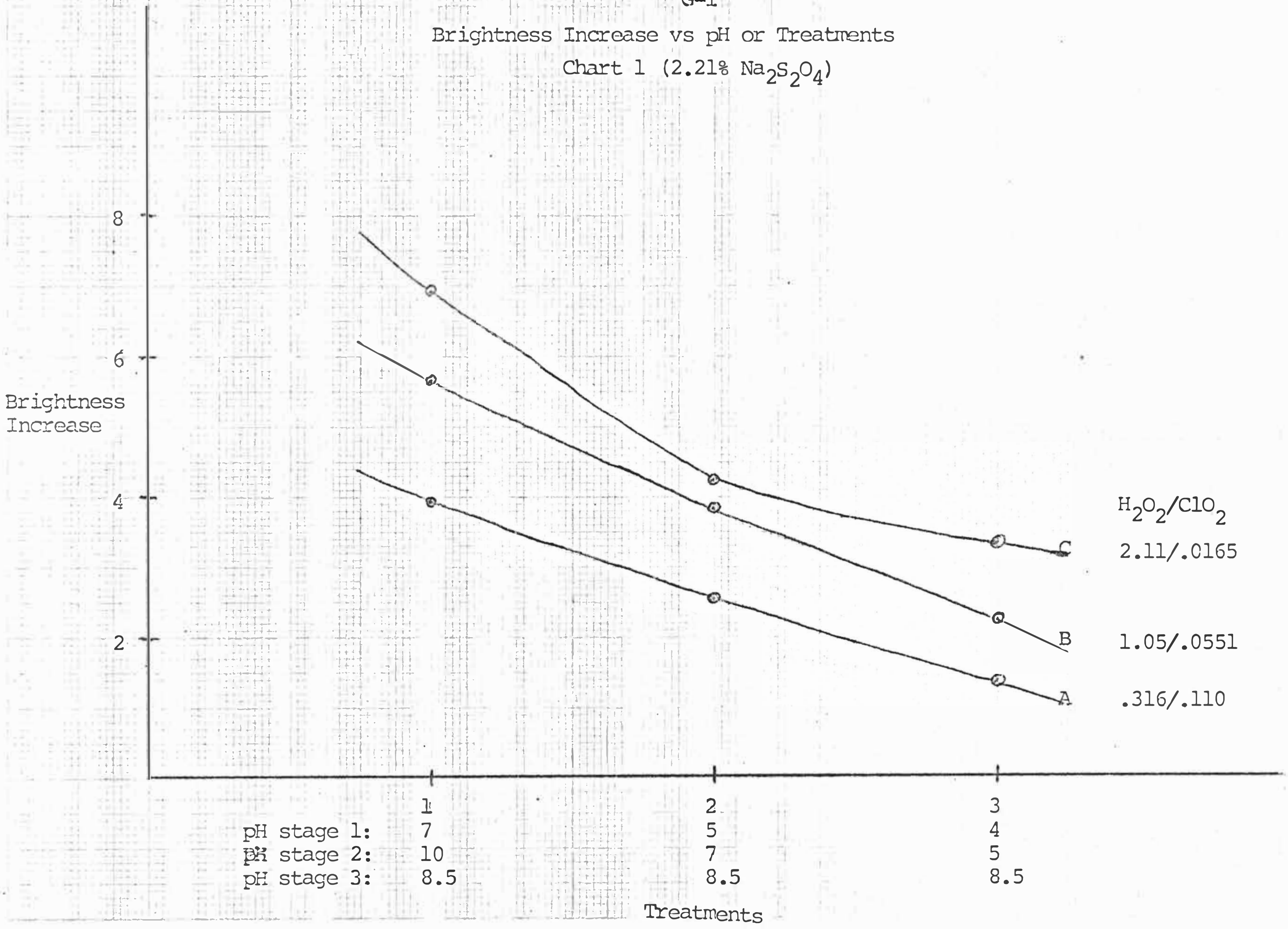
Values given as % act. Cl_2

* Values are the average of the two trial runs.

G-1

Brightness Increase vs pH or Treatments

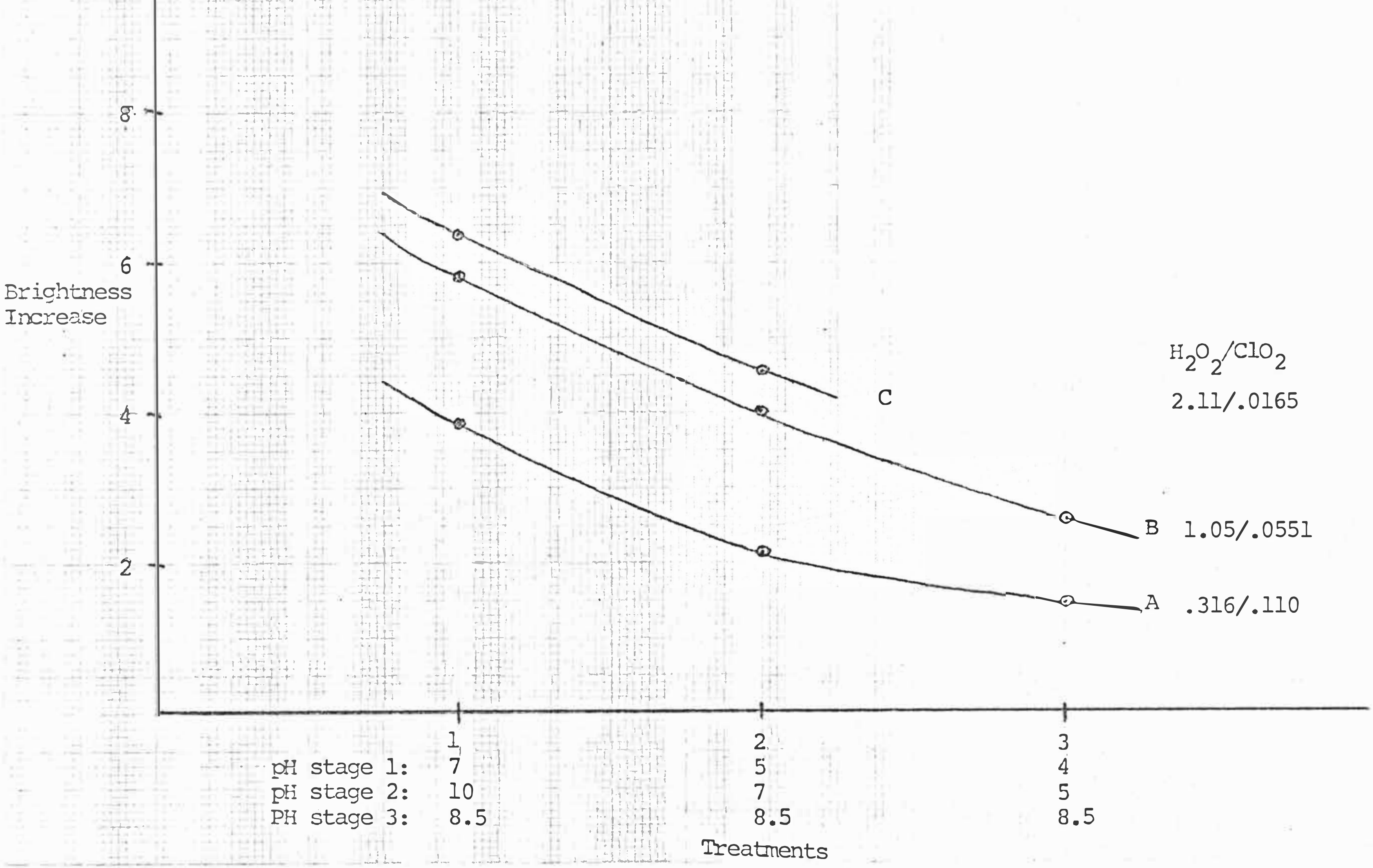
Chart 1 (2.21% $Na_2S_2O_4$)



G-2

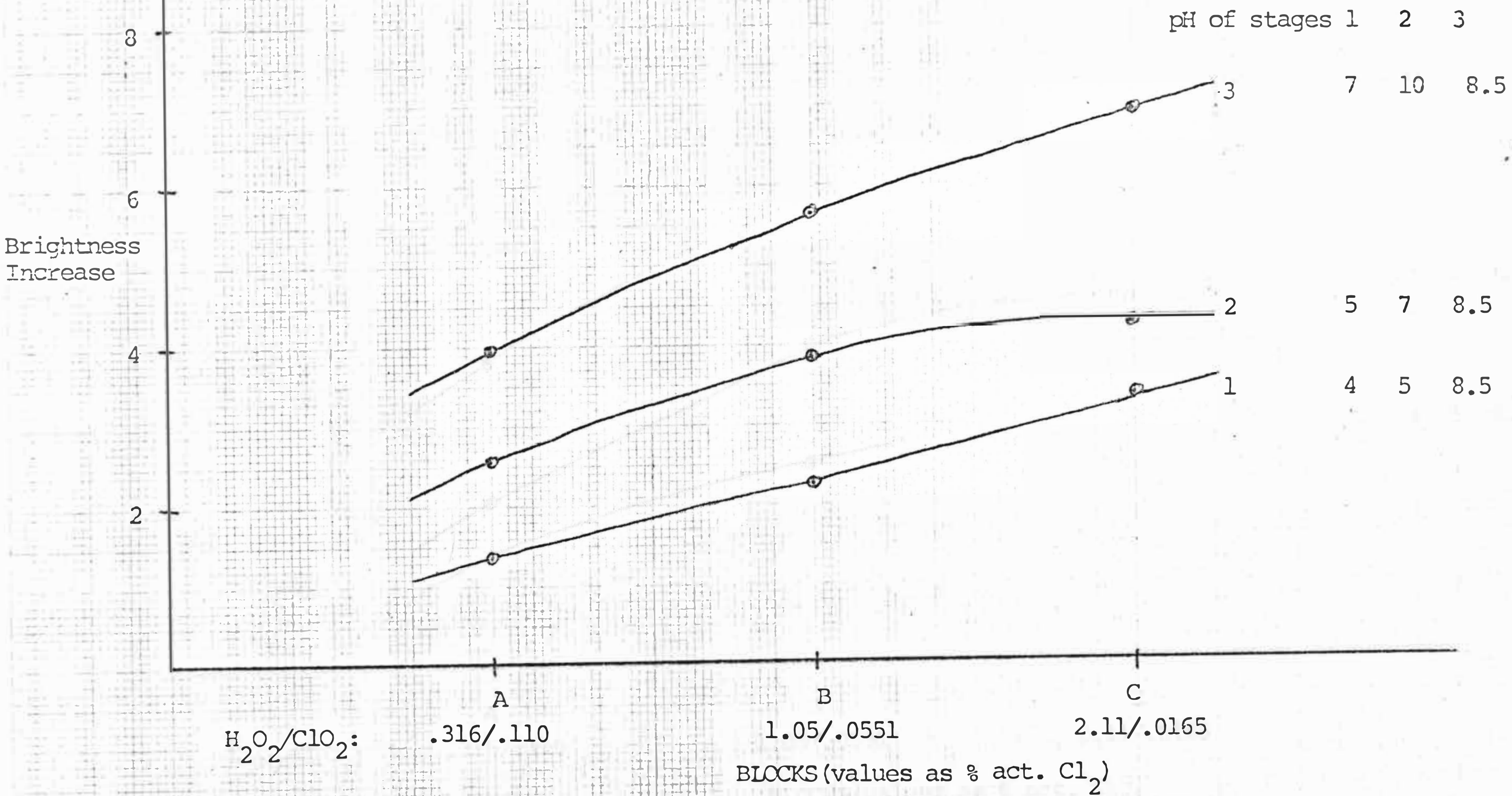
Brightness Increase vs pH or Treatments

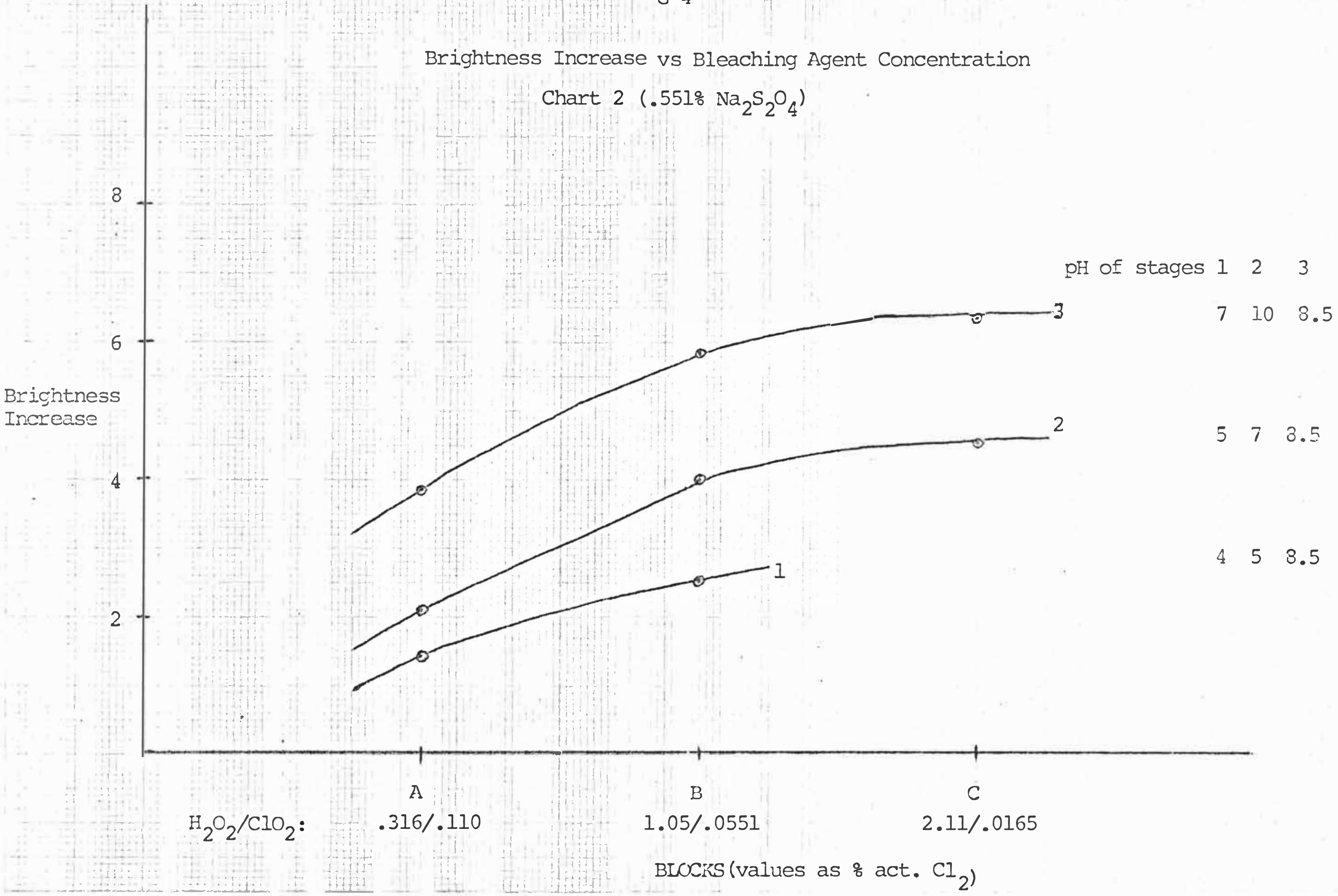
Chart 2 (.551% $Na_2S_2O_4$)



Brightness Increase vs Bleaching Agent Concentration

Chart 1 (2.21% $Na_2S_2O_4$)





Brightness Increase vs Bleaching Agent Concentration

Chart 2 (.551% $Na_2S_2O_4$)

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APPENDIX A

Flow Chart of Pulp Through Pilot Plant Recycling Unit

A) Handsort paper stock; removed all colored inserts and foreign objects.

B) Weighed out;

46 lbs.	newsprint
114 lbs.	IBM computer stock
114 lbs.	office bond
274 lbs.	total based on B.D. fiber

C) Paper stock into hydropulper; temperature 65°C, consistancy 6%, time 40 min.. Then diluted to 2.5%.

D) Belcor unit at a 2.5% consistancy. Some reject pulp screened by Johnson screen.

E) Finchk screen; slots at .012" were used, consistancy was .7%. Accepts showed bouyant black particles. Rejects showed light density particles, mainly styrafoam granules.

F) Bird cyclone cleaners; four primary and one secondary at .7% consistancy. Rejects produced dirt, staples, strings, stones, and grit.

G) Bergstrom reverse cleaners; used a .7% consistancy. Rejects produced light density and heavy particles.

H) Side hill washer; using 80 mesh screen and one pass. Wash water indicated loss of fiber fines, inks, and coating particles.

I) Six grams of slimacide V-10 from Vineland Chemical Co. was added after side hill washer with agitation.

J) Screw press; dewatered pulp from 5% to 16% consistancy.

K) Pulp stored in polyethylene bags at a temperature of approx. 45°F.

APPENDIX B

Differences Between Brightness & Opacity Values of Duplicate Runs

Chart 1 (2.21% $Na_2S_2O_4$)

	1	2	3
A	0.3	0.0	0.1
B	0.6	0.5	0.3
C	0.4	0.8	2.2

Brightness

	1	2	3
A	1.9	0.8	0.9
B	2.0	2.6	1.4
C	1.1	0.6	0.5

Opacity

Chart 2 (.551% $Na_2S_2O_4$)

1	2	3
0.7	0.2	0.5
1.4	0.1	0.5
2.4	0.6	1.0

Brightness

	1	2	3
A	1.6	2.1	0. 3
B	0.4	0.2	0.7
C	2.6	0.6	0.7

Opacity

Treatment	1	2	3
H stage 1	7	5	4
H stage 2	10	7	5
H stage 3	8.5	8.5	8.5

Block	A	B	C
H_2O_2/ClO_2	.316/.110	1.05/.0551	2.11/.0165

Values given as % act. Cl_2